



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1659a

Methane in Air

(Nominal Amount-of-Substance Fraction – 10 $\mu\text{mol/mol}$)

This certificate reports the certified values for Lot 11-G-XX.

This Standard Reference Material (SRM) is a primary gas mixture for which the amount-of-substance fraction (expressed as concentration [1]), may be related to secondary working standards. The SRM is intended for the calibration of instruments used for methane determinations and for other uses.

This SRM mixture is supplied in a DOT 3AL specification aluminum (6061 alloy) cylinder with a water volume of 6 L. Mixtures are shipped with a nominal pressure exceeding 12.4 MPa (1800 psi), which provides the user with 0.73 m³ (25.8 ft³) of useable mixture. The cylinder is the property of the purchaser and is equipped with a CGA-590 brass valve, which is the recommended outlet for this methane mixture. NIST recommends that this cylinder **NOT** be used below 0.7 MPa (100 psi).

Certified Value: This SRM mixture has been certified for methane concentration. The certified value, given below, applies to the identified cylinder and NIST sample number.

Methane Concentration: 9.863 $\mu\text{mol/mol}$ \pm 0.030 $\mu\text{mol/mol}$

Cylinder Number: SAMPLE

NIST Sample Number: SAMPLE

The uncertainty of the certified value includes the estimated uncertainties in the NIST standards, the analytical comparisons to the lot standard (LS), and the uncertainty of comparing the LS with each of the mixtures comprising the lot. The uncertainty is expressed as an expanded uncertainty $U = ku_c$ with u_c determined by experiment and a coverage factor of $k = 2$. The true value for the methane amount-of-substance is asserted to lie in the interval defined by the certified value $\pm U$ with a level of confidence of approximately 95 % [2].

Expiration of Certification: This certification is valid until **25 April 2012**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification will be nullified if the SRM is contaminated or modified. NIST will monitor this SRM over the period of its certification. If changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Hydrotest Date: 04/02

Blend date: 10/02

Cylinder and Gas Handling Information: NIST recommends the use of a high purity, two-stage pressure regulator with a stainless steel diaphragm and CGA-590 outlet to safely reduce the pressure and to deliver this SRM mixture to the instrument. The regulator should be purged several times to prevent accidental contamination of the sample.

The analytical measurements leading to the certification of this current SRM lot were performed by M.E. Kelley of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 19 June 2006
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

The overall direction and coordination of the technical work required for certification of this SRM lot were performed by F.R. Guenther of the NIST Analytical Chemistry Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Mixture Preparation: The gas mixtures comprising this SRM lot were prepared in accordance with NIST technical specifications by a commercial specialty gas vendor under contract to NIST. The specifications stipulate that each SRM mixture be identical in methane concentration and stable with time.

Analytical Methods: Analyses of the methane concentration for this lot of cylinders were conducted by inter-comparing each cylinder mixture to a representative cylinder chosen from the lot, the lot standard (LS), using gas chromatography with a flame ionization detector (GC/FID). Assignment of the methane concentration to the LS was accomplished by inter-comparison to primary gravimetric standards using GC/FID.

Homogeneity Analysis: Each of the methane mixtures that comprise this SRM lot was compared to the LS using GC/FID. An analysis of variance indicated that sample-to-sample methane concentration differences were statistically insignificant. This indicates that within the precision of the NIST measurements, all of the cylinders comprising this SRM lot have identical methane concentrations. Therefore, one concentration has been assigned to the entire SRM lot.

Methane Concentration Value Assignment: The certified methane concentration for this SRM lot was computed from the assigned concentration for the lot standard and the homogeneity analysis.

Stability: Periodic analyses of SRM units from this lot are performed at NIST to monitor stability. If significant changes in the methane concentration are observed, the purchaser will be notified. Refer to the "Cylinder and Gas Handling Information" section for proper handling of this SRM.

Other Analyses: Additional analyses performed during the certification procedure are given below. The concentrations reported are **NOT** certified values. These values are given for informational purposes only.

Constituent	Concentration
Argon	0.93 % mol/mol
Carbon Dioxide	369 µmol/mol
Oxygen	20.9 % mol/mol
Total Other Hydrocarbons	0.02 µmol/mol (expressed as methane)

REFERENCES

- [1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811, (1995).
- [2] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed., International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.

Certificate Revision History: 19June 2006 (Extension of the expiration date and minor editorial changes); 09 October 2003 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.